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UNITED STATES GOVERNMENT SPECIFICATION FOR IRON-OXIDE AND IRON-HYDROXIDE PAINTS

FEDERAL SPECIFICATIONS BOARD

STANDARD SPECIFICATION No. 13

This Specification was officially adopted by the Federal Specifications Board, on February 3, 1922, for the use of the Departments and Independent Establishments of the Government in the purchase of materials covered by it.

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1. GENERAL

This specification applies to iron-oxide and iron-hydroxide paints of red and brown colors. The paint may be ordered in the form of either semipaste paint or ready-mixed paint.

The basis of purchase may be either by net weight or by volume (231 cubic inches to the gallon).

(*a*) PIGMENT.—The pigment in both semipaste and ready-mixed paint shall be very finely ground iron oxide, iron hydroxide, siliceous minerals or a mixture thereof, to which carbon pigment may be added, if necessary, to produce the required color. It must be free from organic coloring matter (dyes or lakes). The pigment shall show on analysis not less than 30 per cent of ferric

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oxide (Fe_2O_3). The total of the ferric oxide, insoluble siliceous matter, and loss on ignition shall be not less than 90 per cent.

(*b*) LIQUID.—The liquid in semipaste paint shall be entirely pure raw or refined linseed oil; in ready-mixed paint it shall contain not less than 75 per cent pure raw linseed oil, the balance to be combined drier and thinner. The thinner shall be turpentine, volatile mineral spirits, or a mixture thereof.

(*c*) SEMIPASTE.—Semipaste shall be made by thoroughly grinding the pigment with pure raw or refined linseed oil.

The semipaste as received and three months thereafter shall be not caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily with linseed oil, turpentine, or volatile mineral spirits, or any combination of these substances, in all proportions without curdling. The color and hiding power when specified shall be equal to that of a sample mutually agreed upon by buyer and seller. The weight per gallon shall be not less than 13½ pounds. The paste shall consist of:

	Maximum	Minimum
	Per cent	Per cent
Pigment	72.0	68.0
Linseed oil	32.0	28.0
Moisture and other volatile matter	.7	.....
Coarse particles and "skins" (total residue retained on No. 325 screen based on pigment)	3.5	.....

(*d*) READY-MIXED PAINT.—Ready-mixed paints shall be well ground, shall not settle badly or cake in the container, shall be readily broken up with a paddle to a smooth uniform paint of good brushing consistency, and shall dry within 18 hours to a full oil gloss, without streaking, running, or sagging. The color and hiding power when specified shall be equal to those of a sample mutually agreed upon by buyer and seller. The weight per gallon shall not be less than 12 pounds. The paint shall consist of:

	Maximum	Minimum
	Per cent	Per cent
Pigment	57.0	53.0
Liquid (containing at least 75 per cent linseed oil)	47.0	43.0
Water	.5	.....
Coarse particles and "skins" (total residue retained on No. 325 screen based on pigment)	3.5	.....

NOTE.—Deliveries will, in general, be sampled and tested by the following methods, but the purchaser reserves the right to use any additional available information to ascertain whether the material meets the specification.

2. SAMPLING

It is mutually agreed by buyer and seller that a single package out of each lot of not more than 1000 packages shall be taken as representative of the whole. Whenever possible an original unopened container shall be sent to the laboratory, and when this is for any reason not done, the inspector shall determine by thorough testing with a paddle or spatula whether the material meets the requirement regarding caking in the container. He shall then thoroughly mix the contents of the container and draw a sample of not less than 5 pounds of the thoroughly mixed paint, place it in a clean, dry metal or glass container, which it shall nearly fill. The container shall be closed with a tight cover, sealed, marked, and sent to the laboratory for test with the inspector's report on caking.

When requested, a duplicate sample may be taken from the same package and delivered to the seller, and the inspector may take a third sample to hold for test in case of dispute.

3. LABORATORY EXAMINATION—SEMIPASTE

(*a*) CAKING IN CONTAINER.—When an original package is received in the laboratory it shall be weighed, opened, and stirred with a stiff spatula or paddle. The paste must be no more difficult to break up than a normal good grade of semipaste paint. The semipaste shall finally be thoroughly mixed, removed from the container, and the container wiped clean and weighed. This weight subtracted from the weight of the original package gives the net weight of the contents. A portion of thoroughly mixed semipaste shall be placed in a clean container and the portions for the remaining tests promptly weighed out.

(*b*) COLOR.—Place some of the paint on a clean, clear glass plate. Place some of the standard agreed upon beside the sample on the plate, turn the glass over, and compare the colors.

(*c*) WEIGHT PER GALLON.—From the weight of a known volume of the paste calculate the specific gravity, which multiplied by 8.33 gives the weight in pounds per gallon. Any suitable container of known volume may be used for the purpose, but a short cylinder of heavy glass with rounded bottom about 75 mm high and having a capacity of from 125 to 175 cc (a glass cap to keep dust from reagent bottle stopper) is a convenient apparatus for the purpose. The capacity of this vessel is determined to within 1 cc. The paste is packed into it until completely full, the top leveled off smooth with a spatula, and weighed to ± 0.5 g. Sub-

tract the weight of the empty container and divide the remainder by the number of cubic centimeters representing the capacity of the container. The quotient is the specific gravity, which can be thus determined within ± 2 in the second decimal place.

(*d*) MIXING WITH LINSEED OIL.—One hundred grams of the paste shall be placed in a cup, 30 cc linseed oil added slowly, with careful stirring and mixing with a spatula or paddle. The resulting mixture must be smooth and of good brushing consistency.

(*e*) MOISTURE AND OTHER VOLATILE MATTER.—Weigh accurately from 3 to 5 g of the paste into a tarred flat-bottomed dish about 5 cm in diameter, spreading the paste over the bottom. Heat at 105 to 110° C for one hour, cool, and weigh. Calculate loss in weight as percentage of moisture and volatile matter.

(*f*) PERCENTAGE OF PIGMENT.—Weigh accurately about 15 g of the paste into a weighed centrifuge tube. Add 20 to 30 cc of "extraction mixture" (see Reagents), mix thoroughly with a glass rod, wash the rod with more of the extraction mixture, and add sufficient of the reagent to make a total of 60 cc in the tube. Place the tube in the container of a centrifuge, surround with water, and counterbalance the container of the opposite arm with a similar tube or a tube with water. Whirl at a moderate speed until well settled. Decant the clear supernatant liquid. Repeat the extraction twice with 40 cc of extraction mixture and once with 40 cc of ether. After drawing off the ether, set the tube in a beaker of water at about 80° C or on top of a warm oven for 10 minutes, then in an oven at 110 to 115° C for two hours. Cool, weigh, and calculate the percentage of pigment. Grind the pigment to a fine powder, pass through a No. 80 screen to remove any skins, and preserve in a stoppered bottle.

(*g*) PREPARATION OF FATTY ACIDS.—To about 25 g of the paste in a porcelain casserole, add 15 cc of aqueous sodium hydroxide (see Reagents) and 75 cc of ethyl alcohol, mix and heat uncovered on a steam bath until saponification is complete (about one hour). Add 100 cc of water, boil, add an excess of sulphuric acid of specific gravity 1.2 (8 to 10 cc will usually suffice), boil, stir, and transfer to a separatory funnel to which some water has been previously added. Draw off as much as possible of the acid aqueous layer, wash once with water, then add 50 cc of water and 50 cc of ether. Shake very gently with a whirling motion to dissolve the fatty acids in the ether, but not so violently as to form an emulsion. Draw of the aqueous layer and wash the

ether layer with one 15 cc portion of water and then with 5 cc portions of water until free from sulphuric acid. Then draw off the water layer completely. Transfer the ether solution to a dry flask and add 25 to 50 g of anhydrous sodium sulphate. Stopper the flask and let stand with occasional shaking at a temperature below 25° C. until the water is completely removed from the ether solution, which will be shown by the solution becoming perfectly clear above the solid sodium sulphate. Decant this clear solution, if necessary, through a dry filter paper, into a dry 100 cc Erlenmeyer flask. Pass a rapid current of dry air (pass through a $CaCl_2$ tower) into the mouth of the Erlenmeyer flask and heat to a temperature below 75° C on a dry hot plate until the ether is entirely driven off.

NOTE.—It is important to follow all of the details, since ether generally contains alcohol and after washing with water always contains water. It is very difficult to remove water and alcohol by evaporation from fatty acids, but the washing of the ether solution and subsequent drying with anhydrous sodium sulphate removes both water and alcohol. Ether, in the absence of water and alcohol, is easily removed from fatty acids by gentle heat.

The fatty acids prepared as above should be kept in a stoppered flask and examined at once.

(*h*) TEST FOR MINERAL OIL AND OTHER UNSAPONIFIABLE MATTER.—Place 10 drops of the fatty acid (*g*) in a 50 cc test tube, add 5 cc of alcoholic soda (see reagents), boil vigorously for five minutes, add 40 cc of water, and mix; a clear solution indicates that not more than traces of unsaponifiable matter are present. If the solution is not clear, the oil is not pure linseed oil.

(*i*) IODINE NUMBER OF FATTY ACIDS.—Place a small quantity of the fatty acids (*g*) in a small weighing burette or beaker. Weigh accurately. Transfer by dropping about 0.15 g (0.10 to 0.20 g) into a 500 cc bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine amount of sample used. Add 10 cc of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc of chloroform to two empty bottles like that used for the sample. Add to each bottle 25 cc of the Hanus solution (see reagents) and let stand with occasional shaking for one-half hour. Add 10 cc of the 15 per cent potassium iodide solution and 100 cc of water, and titrate with standard sodium thiosulphate, using starch as indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titrations and the

titration on the sample and the iodine value of the thiosulphate solution, calculate the iodine number of the sample tested. (Iodine number is centigrams of iodine to 1 g of sample.) If the iodone number is less than 170, the oil does not meet the specification.

(*j*) Coarse Particles and Skins.—Dry in an oven at 105 to 110° C a No. 325 screen, cool, and weigh accurately. Weigh an amount of semipaste containing 10 g of pigment (see 3 (*f*)), add 50 cc of kerosene, mix thoroughly, and wash with kerosene through the screen, breaking up all lumps, but not grinding. After washing with kerosene until all but the particles too coarse to pass the screen have been washed through, wash all kerosene from the screen with ether or petroleum ether, heat the screen for one hour at 105 to 110° C, cool, and weigh.

4. ANALYSIS OF PIGMENT

(*a*) Organic Coloring Matter.—(A. S. T. M. Standards, 1918, p. 656). Test the pigment successively with hot water, 95 per cent alcohol, alcoholic sodium hydroxide, and acetic acid. Chloroform, sodium hydroxide, sulphuric acid, hydrochloric acid-stannous chloride solution, and other reagents may be tried. The solutions should remain colorless. The presence of an organic color may often be detected by the characteristic odor given off on ignition.

(*b*) Total Iron Oxide.—Ignite 1 g of the pigment in a porcelain crucible at a dull red heat to destroy organic matter. Transfer to a 500 cc Erlenmeyer flask and add 20 cc of 1:1 hydrochloric acid. Digest just short of boiling till no dark specks can be seen in the insoluble residue. When the residue is light in color, the solution of iron may be considered complete. Dilute to 100 cc and without filtering, add 3 g of granulated zinc; put a funnel in the neck of the flask and heat when the action slackens; if basic iron salts separate out, add a few drops of hydrochloric acid. When the reduction is complete, add 30 cc of sulphuric acid (1:2), and as soon as the residual zinc is dissolved, wash down the funnel and neck of the flask with a fine jet of water. Now add 200 cc of cool water and 30 cc of titrating solution (see reagents) and titrate with standard potassium permanganate. Run a blank on the zinc and calculate iron as Fe_2O_3. Any other accurate method for determining iron may be used at the option of the analyst.

(*c*) Loss on Ignition.—Ignite 1 g of the pigment to constant weight. It is safest to use a porcelain crucible for this purpose.

(*d*) INSOLUBLE SILICEOUS MATTER.—Transfer 1 g of the pigment to a porcelain dish, ignite at a dull red heat to destroy organic matter, cool, add 20 cc of hydrochloric acid, 1:1, cover and heat on a steam bath until no dark specks can be seen in the insoluble residue. Remove cover, add 10 cc of strong hydrochloric acid, evaporate to dryness on a steam bath, moisten with hydrochloric acid, add water, and wash on to a filter paper with hot water; wash, ignite, and weigh the insoluble siliceous matter.

5. LABORATORY EXAMINATION—MIXED PAINT

(*a*) CAKING IN CONTAINER.—Follow the procedure outlined in 3(*a*), noting that the paint should be no more difficult to break up than a good grade of mixed paint.

(*b*) COLOR.—Follow the procedure outlined in 3(*b*).

(*c*) WEIGHT PER GALLON.—Weigh a clean, dry, 100 cc graduated flask. Fill to the mark with the thoroughly mixed paint and weigh again. The increase in weight expressed in grams, divided by 100, gives the specific gravity, which multiplied by 8.33 gives the weight in pounds per gallon.

(*d*) BRUSHING PROPERTIES AND TIME OF DRYING.—Brush the well-mixed paint on a suitable panel, which may be ground glass, steel, or well-filled wood. Note whether the paint works satisfactorily under the brush. Place the panel in a vertical position in a well-ventilated room and let stand for 18 hours. The paint shall be dry, smooth, and free from streaks.

(*e*) WATER.—Mix 100 g of the paint in a 300 cc flask with 75 cc of toluol. Connect with a condenser and distill until about 50 cc of distillate has been collected in a graduate. The temperature in the flask should be then about 105 to 110° C. The number of cubic centimeters of water collecting under the toluol in the receiver is the percentage of water in the paint. Material complying with the specification should yield less than 0.5 cc.

(*f*) VOLATILE THINNER.—Follow the procedure outlined in 3(*e*). Correct the result for any water found (see 5(*e*)) and report the remainder as volatile thinner.

(*g*) PERCENTAGE OF PIGMENT.—Follow the procedure outlined in 3(*f*).

(*h*) TESTING NONVOLATILE VEHICLE.—Follow the procedure outlined in 3(*g*), 3(*h*), and 3(*i*), except that in the preparation of the fatty acids the mixture of paint and alkali is heated on the steam bath until all volatile thinner is driven off.

(*i*) COARSE PARTICLES AND SKINS.—Follow the procedure outlined in 3(*j*).

(*j*) TESTING PIGMENT.—Follow the procedure outlined in 4(*a*) to 4(*d*), inclusive.

6. REAGENTS

(*a*) EXTRACTION MIXTURE.—

10 volumes ether (ethyl ether).
6 volumes benzol.
4 volumes methyl alcohol.
1 volume acetone.

(*b*) AQUEOUS SODIUM HYDROXIDE.—Dissolve 100 g of sodium hydroxide in distilled water and dilute to 300 cc.

(*c*) STANDARD SODIUM THIOSULPHATE SOLUTION.—Dissolve pure sodium thiosulphate in distilled water (that has been well boiled to free it from carbon dioxide) in the proportion of 24.83 g of crystallized sodium thiosulphate to 1000 cc of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Treadwell-Hall, Analytical Chemistry, vol. 2, 3d ed., p. 646.) This solution will be approximately decinormal, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly decinormal. Preserve in a stock bottle provided with a guard tube filled with soda lime.

(*d*) STARCH SOLUTION.—Stir up 2 to 3 g of potato starch or 5 g of soluble starch with 100 cc of 1 per cent salicylic acid solution, add 300 to 400 cc boiling water, and boil the mixture until the starch is practically dissolved, then dilute to 1 liter.

(*e*) POTASSIUM IODIDE SOLUTION.—Dissolve 150 g of potassium iodide free from iodate in distilled water and dilute to 1000 cc.

(*f*) HANUS SOLUTION.—Dissolve 13.2 g of iodine in 1000 cc of 99.5 per cent glacial acetic acid, which will not reduce chromic acid. Add enough bromine to double the halogen content, determined by titration (3 cc of bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromine is added.

(*g*) ALCOHOLIC SODIUM HYDROXIDE SOLUTION.—Dissolve pure sodium hydroxide in 95 per cent ethyl alcohol in the proportion of about 22 g per 1000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle and keep well stoppered. This solution should be colorless or only slightly yellow when used,

and it will keep colorless longer if the alcohol is previously treated with sodium hydroxide (about 80 g to 1000 cc), kept at about 50° C for 15 days, and then distilled.

(*h*) POTASSIUM PERMANGANATE SOLUTION.—Dissolve 5.7 g of pure potassium permanganate in a liter of distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize as follows: In a 400 cc beaker dissolve 0.40–0.50 g of Bureau of Standards' sodium oxalate in 250 cc of hot water (80 to 90° C) and add 15 cc of dilute sulphuric acid (1 : 1). Titrate at once with the potassium permanganate solution, *stirring the liquid vigorously and continuously*. The permanganate must not be added more rapidly than 10 to 15 cc per minute, and the last 0.5 to 1 cc must be added dropwise with particular care to allow each drop to be fully decolorized before the next is introduced. The solution should not be below 60° C by the time the end point is reached. (More rapid cooling may be prevented by allowing the beaker to stand on a small asbestos-covered hot plate during the titration. The use of a small thermometer as a stirring rod is most convenient.) The weight of sodium oxalate used multiplied by 0.8334 gives its iron equivalent. The permanganate solution should be kept in a glass-stoppered bottle painted black to keep out light.

(*i*) TITRATING SOLUTION.—Dissolve 160 g of manganese sulphate in water, dilute to 1750 cc, add 330 cc of orthophosphoric acid (specific gravity 1.72), and 320 cc of concentrated sulphuric acid.

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